

DECLARATION UNDER 37 CFR 1.132

Commissioner for Patents  
Washington, D.C. 20231

SIR:

I, Toshiya KITAMURA, declare as follows:

- 1 I am an inventor of the above-identified patent application.
- 2 I graduated from Tokyo University of Science in 1993, and I received the degree of Master of Engineering.
- 3 I have worked for Dowa Mining Co., Ltd. of Tokyo, Japan since 1993, and I presently hold the position of Group Leader of Zinc Powder Group.
4. The following tests were carried out under my supervision, and the arguments were prepared by me.
5. Starting at page 3, line 6 from the bottom in the last Office Action the Examiner rejects claims 1, 2, 7, 8 and 14 under 35 USC 102 (b) as being anticipated by Miyasaka, JP-5-151968.

Starting at page 4, line 12 in the same Office Action the Examiner rejects claim 6 under 35 USC 102(b)/103(a) as being anticipated by, and alternatively unpatentable over, Miyasaka, JP 5-151968. However, I neither believe that claims 1, 2, 7, 8 and 14 are anticipated by Miyasaka, JP-5-151968 nor do that claim 6 is unpatentable over Miyasaka, JP 5-151968. The reason is as follows.

The following tests were carried out for determining the ability of bismuth to suppress the evolution of hydrogen gas when bismuth is attached to the surfaces of zinc particles by means of ionic substitution according to the invention of Miyasaka, JP-5-151968.

Experiment 1

A powder of an alloyed zinc with a particle size in the range of 75~850  $\mu$ m and having the composition consisting of 92 ppm by weight (hereunder simply referred to by "ppm") bismuth ( Bi ), 30 ppm aluminum ( Al ), 495 ppm indium ( In ) and the balance of zinc ( Zn ) was prepared by the process called atomization.



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zinc powder compo  
different*

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35 parts by weight of an electrolyte consisting of 40 wt% KOH solution saturated with zinc oxide was added to 65 parts by weight of the alloyed zinc powder mentioned above and the resulting mixture was kept at 60 °C for a predetermined period of time. The amount of hydrogen gas evolution was determined by calculation in terms of  $\mu\text{l/g} \cdot \text{day}$  based on the amount of gas evolved in the third day from the beginning of experiment. Five times of experiments were repeated in the same manner as in the first run except that each time another 65 parts by weight of the same alloyed zinc powder was used. The results are as shown in Table 1 below.

**Table 1**

Amount of Bi adhesion	Amount of hydrogen gas evolution ( $\mu\text{l/g} \cdot \text{day}$ )						
	Run No. 1	Run No. 2	Run No. 3	Run No. 4	Run No. 5	Average	Standard Deviation
(0 ppm)	12.7	12.0	11.3	13.5	16.2	13.1	1.9

### **Experiment 2**

1 kg of the same alloyed zinc powder as prepared in Experiment 1 was put into 500 ml of an aqueous 1 wt% sulfuric acid solution, to which was added, bit by bit with agitation, 50 ml of an aqueous 1 wt% nitric acid solution containing 1 mg/ml of bismuth so as to make bismuth uniformly deposit on the surfaces of the alloyed zinc particles making use of the difference in ionization tendencies of zinc and bismuth. Then, the bismuth-coated zinc particles were separated by filtration, washed with water, dehydrated and dried. 35 parts by weight of an electrolyte consisting of 40 wt% KOH solution saturated with zinc oxide was added to 65 parts by weight of the bismuth (50 ppm)-coated alloyed zinc powder and the resulting mixture was kept at 60 °C for a predetermined period of time. The amount of hydrogen gas evolution was determined by calculation in terms of  $\mu\text{l/g} \cdot \text{day}$  based on the amount of gas evolved in the third day from the beginning of experiment. Five runs of experiments were repeated in the same manner as in the first run explained above except that each time another 35 parts by weight of the same bismuth (50 ppm)-coated alloyed zinc powder was used. The results are as shown in Table 2 below.

**Table 2**

Amount of Bi-coating (50 ppm)	Run No. 1	Run No. 2	Run No. 3	Run No. 4	Run No. 5	Average	Standard Deviation
Amount of hydrogen gas evolution ( $\mu$ l/g · day)	31.3	31.3	26.0	22.6	30.5	28.3	3.9

### **Experiment 3**

The same procedures were repeated as in Experiment 2 except that the amount of bismuth adhesion was changed from 50 ppm to 100 ppm by using 100 ml of an aqueous 1 wt% nitric acid solution having bismuth content of 1 mg/ml rather than 50 ml of the same aqueous nitric acid solution. The amount of hydrogen gas evolution was also determined in the same manner as in Experiment 2 by calculation in terms of  $\mu$ l/g · day based on the amount of gas evolved in the third day from the beginning of experiment. Five runs of experiment were also repeated in the same manner as in the case of Experiment 2. The results are as shown in Table 3 given below.

**Table 3**

Amount of Bi-coating (100 ppm)	Run No. 1	Run No. 2	Run No. 3	Run No. 4	Run No. 5	Average	Standard Deviation
Amount of hydrogen gas evolution ( $\mu$ l/g · day)	13.3	23.3	20.0	16.3	15.9	17.8	3.9

### **Experiment 4**

The same procedures were repeated as in Experiment 3 except that the amount of bismuth adhesion was changed from 100 ppm to 300 ppm by using 100 ml of an aqueous 1 wt% nitric acid solution having bismuth content of 3 mg/ml rather than 100 ml of the aqueous nitric acid solution having the bismuth content of 1 mg/ml. The amount of hydrogen gas evolution was also determined in the same manner as in Experiment 3 by calculation in terms of  $\mu$ l/g · day based on the amount of gas evolved in the third day from the beginning of experiment. Five runs of experiment were also repeated in the same manner

as in the case of Experiment 3. The results are as shown in Table 4 given below.

**Table 4**

Amount of Bi-coating (300 ppm)	Run No. 1	Run No. 2	Run No. 3	Run No. 4	Run No. 5	Average	Standard Deviation
Amount of hydrogen gas evolution ( $\mu$ l/g $\cdot$ day)	16.7	16.0	19.3	18.2	12.7	16.6	2.5

#### **Experiment 5**

The same procedures were repeated as in Experiment 4 except that the amount of bismuth adhesion was changed from 300 ppm to 500 ppm by using 167 ml of an aqueous 1 wt% nitric acid solution having the bismuth content of 3 mg/ml rather than 100 ml of the aqueous nitric acid solution having the bismuth content of 1 mg/ml. The amount of hydrogen gas evolution was also determined in the same manner as in Experiment 3 by calculation in terms of  $\mu$  l/g  $\cdot$  day based on the amount of gas evolved in the third day from the beginning of experiment. Five runs of experiment were also repeated in the same manner as in the case of Experiment 3. The results are as shown in Table 4 given below.

**Table 5**

Amount of Bi-coating (500 ppm)	Run No. 1	Run No. 2	Run No. 3	Run No. 4	Run No. 5	Average	Standard Deviation
Amount of hydrogen gas evolution ( $\mu$ l/g $\cdot$ day)	12.2	10.9	15.6	16.2	11.8	13.3	2.4

#### **Experiment 6**

The same procedures were repeated as in Experiment 5 except that the amount of bismuth adhesion was changed from 500 ppm to 1000 ppm by using 333 ml of an aqueous 1 wt% nitric acid solution having the bismuth content of 3 mg/ml rather than 167 ml of the aqueous nitric acid solution having the bismuth

content of 3 mg/ml. The amount of hydrogen gas evolution was also determined in the same manner as in Experiment 4 by calculation in terms of  $\mu$  l/g  $\cdot$  day based on the amount of gas evolved in the third day from the beginning of experiment. Five runs of experiment were also repeated in the same manner as in the case of Experiment 5. The results are as shown in Table 6 given below.

**Table 6**

Amount of Bi-coating (1000 ppm)	Run No. 1	Run No. 2	Run No. 3	Run No. 4	Run No. 5	Average	Standard Deviation
Amount of hydrogen gas evolution ( $\mu$ l/g $\cdot$ day)	20.2	18.9	21.3	16.5	16.5	18.7	2.2

The results of the above six experiments are collectively shown in the following Table 7.

**Table 7**

Amount of Bi-coating	Amount of gas evolution	
	Average ( $\mu$ l/g $\cdot$ day)	Standard Deviation ( $\mu$ l/g $\cdot$ day)
Experiment 1 ( 0 ppm )	13.1	1.9
Experiment 2 ( 50 ppm )	28.3	3.9
Experiment 3 ( 100 ppm )	17.8	3.9
Experiment 4 ( 300 ppm )	16.6	2.5
Experiment 5 ( 500 ppm )	13.3	2.4
Experiment 6 ( 1000 ppm )	18.7	2.1

#### **Experiment 7**

An alloyed zinc powder having a composition consisting of 89 ppm Bi, 31.1

ppm Al, 516 ppm In and the balance of Zn with a particle size in the range of 75 to 850  $\mu$  m was prepared by the process called atomization. A proper amount corresponding to 50 ppm, based on the weight of the alloyed zinc, of bismuth powder available from Saitama Dowa High-tech Co., Ltd. (99% up purity) was dry mixed with the alloyed zinc powder prepared above. The resulting mixture was subjected to the hydrogen gas evolution test according to the same procedures as described in Experiment 1. Namely, 35 parts by weight of an electrolyte consisting of 40 wt% KOH solution saturated with zinc oxide was added to 65 parts by weight of the alloyed zinc powder with which 50 ppm by weight of bismuth was dry-mixed and the Bi (50 ppm)- dry-mixed alloyed zinc powder was kept at 60 °C for a predetermined period of time. The amount of hydrogen gas evolution was determined in the same manner as in Experiment 1 by calculation in terms of  $\mu$  l/g  $\cdot$  day based on the amount of gas evolved in the third day from the beginning of experiment. Five times of experiments were repeated in the same manner as in the first run except that each time another 35 parts by weight of the same alloyed zinc powder was used. The results are collectively shown in Table 8 below together with the results of Experiments of 8 – 11.

#### Experiments of 8 – 11

The same procedures as in Experiment 7 were repeated except that the amount of Bi was changed from 50 ppm to 100, 300, 500 and 1000 ppm, respectively. The results of these experiments are collectively shown in Table 8.

**Table 8**

	Amount of metallic bismuth powder dry-mixed with the alloyed zinc powder according to the present invention ( ppm )				
	50	100	300	500	1000
No. 1	13.9	12.2	5.3	3.0	4.7
No. 2	15.8	8.5	6.5	3.0	5.2
No. 3	11.7	7.8	4.6	5.4	2.9
No. 4	12.8	10.2	4.6	8.2	8.4
No. 5	15.0	11.3	9.4	2.5	5.9
Average	14.4	10.0	6.0	4.4	5.4
Standard Deviation	1.6	1.8	2.1	2.4	2.0

I hereby declare that all statements made herein of my own knowledge are

true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date 25. Nov. 2002 By Toshiya Kitamura